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APPLICATION NUMBER: *60/415,005*
FILING DATE: *October 01, 2002*
RELATED PCT APPLICATION NUMBER: *PCT/US03/31263*

By Authority of the
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Attorney Docket No. P35458 (070132.0176)
Express Mail Label No: ET346773375US

10/01/02 JC682 U.S. PTO

JC682 U.S. PTO 60/415005

PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53(c).

INVENTOR(S)		
Given Name (first and middle [if any])	Family Name or Surname	Residence (City and either State or Foreign Country)
William	Neuberg	Perrineville, New Jersey

☐ *Additional inventors are being named on the ___ separately numbered sheets attached hereto*

TITLE OF THE INVENTION (280 characters max)

METHOD FOR INCORPORATING POLYTETRAFLUOROETHYLENE (PTFE) INTO SYNTHETIC SOLUTION SPUN FIBERS TO PRODUCE FIBERS AND TEXTILES HAVING IMPROVED PROPERTIES

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ENCLOSED APPLICATION PARTS *(check all that apply)*

☑ Specification *Number of Pages* 17

☐ Drawing(s) *Number of Sheets*

☐ Application Data Sheet. See 37 CFR 1.76

☐ CD(s), Number

☑ Other (specify) 1 (Abstract)

METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT

☑ Applicant claims small entity status. See 37 CFR 1.27.

☑ A check or money order is enclosed to cover the filing fees

☐ The Commissioner is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number 02-4377

☐ Payment by credit card Form PTO-2038 is attached.

FILING FEE AMOUNT ($) $80

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

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Respectfully submitted,

SIGNATURE Tara E. Agnew

Date: Oct. 1, 2002

TYPED or PRINTED NAME Tara E. Agnew

REGISTRATION NO *(if appropriate)* 50,589

Docket Number P35458 (070132.01

TELEPHONE (212) 408-2654

USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

[redacted]

BAKER BOTTS L.L.P

60[redacted]5005 ,,100102

PROVISIONAL APPLICATION COVER SHEET
Additional Page

Docket Number	P35458 (070132.0176)	Type a plus sign (+) inside this box →	+

INVENTOR(S)/APPLICANT(S)		
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60 5005 .100102

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TOTAL AMOUNT OF PAYMENT	($) 80

Complete if Known	
Application Number	To be assigned
Filing Date	October 1, 2002
First Named Inventor	Neuberg
Examiner Name	--
Group Art Unit	--
Attorney Docket No.	P35458 (070132.0176)

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FEE CALCULATION

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330	165	Design filing fee	
510	255	Plant filing fee	
740	370	Reissue filing fee	
160	80	Provisional filing fee	80

SUBTOTAL (1) ($) 80

2. EXTRA CLAIM FEES

		Extra Claims		Fee from below		Fee Paid
Total Claims	-	20** = 0	X		=	0
Independent Claims	-	3** = 0	X		=	0
Multiple Dependent					=	

Large Entity Fee ($)	Small Entity Fee ($)	Fee Description
18	9	Claims in excess of 20
84	42	Independent claims in excess of 3
280	140	Multiple dependent claim, if not paid
84	42	** Reissue independent claims over original patent
18	9	** Reissue claims in excess of 20 and over original patent

SUBTOTAL (2) ($) 0

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FEE CALCULATION (continued)

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130	65	Surcharge - late filing fee or oath	
50	25	Surcharge - late provisional filing fee or cover sheet	
130	130	Non-English specification	
2,520	2,520	For filing a request for *ex parte* reexamination	
920*	920*	Requesting publication of SIR prior to Examiner action	
1,840*	1,840*	Requesting publication of SIR after Examiner action	
110	55	Extension for reply within first month	
400	200	Extension for reply within second month	
920	460	Extension for reply within third month	
1,440	720	Extension for reply within fourth month	
1,960	980	Extension for reply within fifth month	
320	160	Notice of Appeal	
320	160	Filing a brief in support of an appeal	
280	140	Request for oral hearing	
1,510	1,510	Petition to institute a public use proceeding	
110	55	Petition to revive - unavoidable	
1,280	640	Petition to revive - unintentional	
1,280	640	Utility issue fee (or reissue)	
460	230	Design issue fee	
620	310	Plant issue fee	
130	130	Petitions to the Commissioner	
50	50	Processing fee under 37 CFR 1.17(q)	
180	180	Submission of Information Disclosure Stmt	
40	40	Recording each patent assignment per property (times number of properties)	
740	370	Filing a submission after final rejection (37 CFR § 1.129(a))	
740	370	For each additional invention to be examined (37 CFR § 1.129(b))	
740	370	Request for Continued Examination (RCE)	
900	900	Request for expedited examination of a design application	

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SUBMITTED BY				Complete (if applicable)	
Name (Print/Type)	Tara E. Agnew	Registration No (Attorney/Agent)	50,589	Telephone	(212) 408-2654
Signature	Tara E. Agnew			Date	Oct. 1, 2002

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TO ALL WHOM IT MAY CONCERN:

Be it known that I, WILLIAM NEUBERG, a citizen of the United States, residing in Perrineville, County of Monmouth, State of New Jersey, whose post office address is 30 Bittner Road, Perrineville, New Jersey 08535, have invented an improvement in

METHOD FOR INCORPORATING POLYTETRAFLUOROETHYLENE (PTFE) INTO SYNTHETIC SOLUTION SPUN FIBERS TO PRODUCE FIBERS AND TEXTILES HAVING IMPROVED PROPERTIES

of which the following is a

SPECIFICATION

CROSS-REFERENCE TO RELATED APPLICATION

[0001] This application claims priority to U.S. Provisional Application Serial Number 60/364,565, filed March 14, 2002.

FIELD OF THE INVENTION

[0002] The present invention generally relates to a method for incorporating highly dispersible polytetrafluoroethylene (PTFE) powder into synthetic solution spun fibers so that the resulting fibers have improved properties generally associated with PTFE, including, for example, low coefficient of friction, improved wear resistance, improved stain resistance and improved light stability and UV-light resistance, when compared to conventional solution spun fibers. The present invention further relates to solution spun fibers made by the method described herein and textiles, fabrics, and articles of

manufacture, which comprise the synthetic solution spun fibers disclosed herein where PTFE has been incorporated into the fibers.

BACKGROUND OF THE INVENTION

[0003] In the textile industry, apparel manufacturers and fiber producers are constantly trying to modify the basic composition of each generic type of synthetic fiber, both chemically and physically, in order to produce fiber variations which provide softer feel, greater comfort, brighter and longer lasting colors, better warmth or cooling, better moisture transport or wicking, and better blending properties when blending with other fibers. "FabricLink, Fabric University – Fabric Producers and Trademarks," <http://www.fabriclink.com/Producers.html>. Thus, a constant need has always existed in the art of fiber production for new and innovative ways to improve the properties of synthetic fibers.

[0004] Various manufacturing processes are known in the art for making synthetic fibers. Many synthetic fibers are created by extrusion, whereby a thick viscous precursor or composition is forced through the tiny holes of a spinneret to form continuous filaments of semi-solid polymer. As the filaments emerge from the holes of a spinneret, the liquid polymer is converted first to a rubbery state and then is solidified. The process of extruding and solidifying filaments is generally known as spinning. One common method of spinning filaments of manufactured or synthetic fibers is generally known as "solution spinning" or "wet spinning."

[0005] Typically, solution spinning processes are used with fiber-forming substances that have been dissolved in a solvent. The spinnerets forming the filaments are submerged in a chemical bath, and as the filaments emerge from the spinnerets, they precipitate out of

the solution and solidify. Synthetic fibers that are typically produced by solution spinning processes include rayon fibers, acrylic fibers, aramid fibers, such as Kevlar® para-aramid fibers, modacrylic fibers, and spandex fibers, among others.

[0006] Rayon, one of the most common synthetic fibers produced by solution spinning, is typically defined as a manufactured fiber composed of regenerated cellulose in which substituents have replaced not more than 15% of the hydrogens of the hydroxyl groups. Generally, during rayon production, purified cellulose is chemically converted into a soluble compound, and a solution of this compound is passed through the spinneret to form soft filaments that are then converted or "regenerated" into almost pure cellulose. Because of this reconversion of the soluble compound into cellulose, rayon is generally referred to as a regenerated cellulose fiber.

[0007] Additives may be used in association with the fiber-forming substance (such as cellulose) prior to extrusion in order to improve the quality of the resulting solution spun fibers. Until the present invention, however, it has not been shown that the addition of polytetrafluoroethylene (PTFE) powder, where the powder is dispersible to low micron or submicron particle size or where the PTFE powder particles have a primary particle size that is low micron or submicron, to the fiber-forming substance for a solution spun fiber may be of great benefit for forming improved solution spun fibers.

[0008] It is generally known that PTFE provides characteristics such as improved slipperiness and non-wettability to materials into which it is incorporated. PTFE is useful when in a powder form or a dispersion form. Dry PTFE powder products are known in the art and are generally available in the industry. Several manufacturers in the fluoropolymer industry produce PTFE powders, and some of these manufacturers

describe the PTFE particle size in their powders as being "submicron" or capable of being dispersed to submicron size.

[0009] A wide array of end uses exists for small particle size or submicron PTFE. For example, small amounts (e.g., about 0.1 to 2% by weight) of powdered PTFE may be incorporated into a variety of compositions to provide the following favorable and beneficial characteristics: (i) in inks, PTFE provides excellent mar and rub resistance characteristics; (ii) in cosmetics, PTFE provides a silky feel; (iii) in sunscreens, PTFE provides increased shielding from UV rays or increased SPF (sun protection factor); (iv) in greases and oils, PTFE provides superior lubrication; and (v) in coatings and thermoplastics, PTFE provides improved abrasion resistance, chemical resistance, weather resistance, water resistance, and film hardness.

[0010] Other, more specific end uses for submicron PTFE powders and dispersions include, but are certainly not limited to: (i) incorporating a uniform dispersion of submicron PTFE particles into electroless nickel coatings to improve the friction and wear characteristics of such coatings (Hadley et al., *Metal Finishing*, **85**:51-53 (December 1987)); (ii) incorporating submicron PTFE particles into a surface finish layer for an electrical connector contact, wherein the PTFE particles provide wear resistance to the surface finish layer (U.S. Patent No. 6,274,254 to Abys et al.); (iii) using submicron PTFE particles in a film-forming binder as a solid lubricant in an interfacial layer, wherein the interfacial layer is part of an optical waveguide fiber (U.S. Patent No. 5,181,268 to Chien); (iv) using a submicron PTFE powder (along with a granulated PTFE powder and TiO_2) in a dry engine oil additive, wherein the additive increases the slip characteristics of the load bearing surfaces (U.S. Patent No. 4,888,122 to McCready); and

(v) combining submicron PTFE particles with autocatalytically-applied nickel/phosphorus for use in a surface treatment system for metals and metal alloys, wherein the PTFE imparts lubrication, low friction, and wear resistance to the resulting surface ("Niflor Engineered Composite Coatings," Hay N., International, Ltd. (1989)). Additional specific examples of end uses for PTFE involve incorporating PTFE into engine oils, using PTFE as a thickener in greases, and using PTFE as an industrial lubricant additive. Willson, *Industrial Lubrication and Tribology*, 44:3-5 (March/April 1992).

[0011] For many applications or end uses incorporating submicron PTFE powders and submicron PTFE dispersions (such as the end uses described above), the beneficial effects being imparted to the application or end use system are derived from the chemical inertness of the PTFE particles and/or the low coefficient of friction of the PTFE particles. In addition, because submicron PTFE particles have such low particle size, they possess a significantly higher ratio of surface area to weight when compared to larger PTFE particles. Thus, submicron PTFE particles (as compared to larger PTFE particles) are better able to supply their useful effects to a desired application system when incorporated at the same weight load.

[0012] In light of the above discussions of the constant evolution in the fiber industry toward making better synthetic fibers having improved properties and the usefulness of PTFE in various applications, it is evident that a need exists in the art for a convenient and inexpensive method by which PTFE, specifically PTFE powder that is dispersible to low micron or submicron particle size, may be incorporated into a given amount of a fiber-forming substance so that the fibers that are spun by a solution spinning process

possess the improved properties associated with PTFE. Furthermore, a need exists for a method of incorporating low micron or submicron PTFE particles uniformly and permanently throughout a solution spun fiber so that textiles, fabrics and clothing formed from the solution spun fibers will not lose, over time, the enhanced properties associated with PTFE because of the wearing away of a surface treatment applied to such a textile, fabric or clothing article. The present invention addresses these and other needs.

SUMMARY OF THE INVENTION

[0013] The present invention relates to a novel method by which polytetrafluoroethylene (PTFE) is incorporated into a synthetic solution spun fiber so that the resulting fiber has many improved properties when compared to conventional solution spun fibers. In the present method, PTFE powder that is dispersible to low micron or submicron particle size is incorporated into the desired fiber-forming substance (such as cellulose) before a fiber or a filament is made by solution spinning. Subsequently, the PTFE-containing fiber-forming substance is solution spun into filaments or fibers, and the resulting filaments or fibers contain PTFE particles dispersed therein. The method of the present invention results in PTFE-enhanced solution spun fibers, wherein PTFE is incorporated directly into the filament and wherein the fibers have the improved properties associated with PTFE. For example, the solution spun fibers resulting from the method of the present invention exhibit a significant decrease in the coefficient of friction when compared to conventional solution spun fibers.

[0014] The use of low micron or submicron particle size PTFE powder as an additive to the fiber-forming substances used to make certain synthetic fibers is important in that the PTFE improves the non-wetting properties of the fibers and textiles made from such

fibers. Thus, fibers incorporating PTFE may be useful in industrial textiles such as textile articles used for filtration and dewatering processes. Such fibers incorporating PTFE may also be used in producing carpets, fabrics for sportswear and outerwear, hot-air balloons, car and plane seats, umbrellas, and the like. Furthermore, the fibers of the present invention may be used to make tightly woven fabrics that are used in parachutes, boat sails, and similar applications. The combination of a tight weave and water shedding may provide a textile for clothing that is both water shedding and breathable. The incorporation of PTFE into such textiles results in other advantages, such as the textile articles being easier to clean.

[0015] The method of the present invention is useful in that the resulting PTFE-enhanced solution spun fibers have many improved properties when compared to conventional synthetic solution spun fibers. Some of these improved properties include but are not limited to the following: lower coefficient of friction; reduced wettability; improved stain resistance; improved washability; improved opacity; enhanced protection from ultraviolet (UV) radiation (which increases the light-fastness and the lifetime of the fiber or fabric); increased color fastness; reduced gas permeability; better abrasion resistance; tighter weave; improved wear index; increased flexibility of the fiber; decreased scroop (where scroop generally refers to sounds of rubbing made by certain fabrics); and lowered amounts of wrinkling when the PTFE-enhanced fibers are incorporated into a fabric or clothing article.

[0016] Additionally, not only does the method of the present invention result in improved solution spun fibers, but the method serves to significantly improve the overall processes by which synthetic fibers are typically made. For example, the increased

slipperiness added to a fiber or filament by the incorporation of PTFE into the fiber-forming substance results in lower production times for fiber production, significantly increased processing speeds, rates of throughput, and overall production rate, longer lifetime of the fiber-making equipment because of the lubricity added by the PTFE, and overall savings in energy expended by the machinery used to form synthetic fibers.

DETAILED DESCRIPTION OF THE INVENTION

[0017] The present invention relates to a method for producing improved solution spun fibers, wherein the fibers are more wear resistant and have a lower coefficient of friction than solution spun fibers that are known in the art. Thus, the method of present invention actually improves the quality of a solution spun fiber by introducing PTFE that is dispersible to low micron or submicron particle size into the fiber-forming substance before the fiber is made by a solution spinning process. The PTFE-enhanced solution spun fibers that are created by the method of the present invention exhibit, among other properties, increased wear resistance, stain resistance, water resistance, and a significantly decreased coefficient of friction, when compared to conventional solution spun fibers known in the art.

[0018] An important objective of the present invention is to incorporate PTFE throughout a solution spun fiber so that the fiber comprises PTFE and experiences the enhanced properties afforded to the fiber by PTFE. This is in comparison to processes and fibers known in the art where PTFE is incorporated only on the surface of solution spun fibers or a fabric made from such fibers and thus can wear away.

[0019] In preferred embodiments of the method of the present invention, the following types of PTFE are useful: PTFE powder that is dispersible to submicron particle size;

PTFE powder that is dispersible to low micron particle size; aqueous or organic dispersions of PTFE powder that is dispersible to submicron particle size; and aqueous or organic dispersions of PTFE powder that is dispersible to low micron particle size. One specific type of PTFE that may be useful in the method of the present invention is the PTFE described in U.S. Provisional Application Serial No. 60/364,565, filed March 14, 2002, the specification of which is hereby incorporated by reference in its entirety.

[0020] In the present application, the designation "submicron particle size" indicates that a given quantity of PTFE powder disperses in isopropyl alcohol (IPA) such that more than 90%, preferably, more than 95%, and more preferably, more than 99% of the PTFE particles have a particle size that is less than 1.00 μm. Furthermore, the designation "low micron particle size" indicates that a given quantity of PTFE powder disperses in isopropyl alcohol (IPA) such that about 95% or more of the PTFE particles have a particle size that is less than 10.00 μm.

[0021] The dispersibility of the PTFE powder down to low micron or submicron sized particles is important because this dispersibility allows for the solution spinning process to not be hindered, for example, by large PTFE particles clogging the spinnerets and making fiber formation difficult. It is envisioned that the method of the present invention allows for PTFE that is dispersible to low micron particle size to be used in higher denier fibers, while PTFE that is dispersible to submicron particle size will be useful for forming both low and high denier fibers.

[0022] The dispersibility of the PTFE particles may be determined by dispersing an amount of PTFE powder in isopropyl alcohol (IPA). Through particle size analysis, the user is given an indication of the mean particle size and the particle size distribution of

the PTFE powder and is thereby able to determine, for example, whether that sample of PTFE powder is 100% dispersible to submicron in size.

[0023] As mentioned above, aqueous or organic dispersions of PTFE that is dispersible either to submicron particle size or low micron particle size may be used in the method of the present invention. PTFE dispersions that are most useful in the present method typically comprise from about 20% to about 60% PTFE by weight. Alternatively, dry PTFE powder that is dispersible either to submicron or low micron particle size may be dispersed directly into the fiber-forming substance (such as cellulose).

[0024] In certain preferred embodiments of the method of the present invention, an aqueous or organic dispersion of PTFE that is dispersible either to submicron particle size or low micron particle size is first provided. The steps for making a PTFE-enhanced solution spun fiber are then employed. For example, the steps for making a PTFE-enhanced rayon fiber are described in detail below.

[0025] The solution spinning process for making rayon fibers involves several steps. Such a process typically begins with cellulose. Specifically, the purified cellulose used for rayon production usually comes from specially processed wood pulp and is sometimes referred to as "dissolving cellulose" or "dissolving pulp" to distinguish it from lower grade pulps used for papermaking and other purposes. Dissolving cellulose is characterized by a high α-cellulose content, meaning that the cellulose comprises long-chain molecules, relatively free from lignin and hemicelluloses or other short-chain carbohydrates.

[0026] Cellulose sheets must first be wet, and this wetting allows for one way of incorporating PTFE according to the present invention. In conventional solution spinning

processes, the cellulose sheets would be wetted with water. However, in one embodiment of the present invention, the cellulose sheets may be wetted with an aqueous dispersion of PTFE that is dispersible either to submicron particle size or low micron particle size. The PTFE is typically incorporated into the cellulose sheets mechanically, for example, by mixing, stirring or blending. As shown below, in alternative embodiments, the PTFE may be incorporated into the cellulose at any point prior to the spinning of the rayon filaments.

[0027] The wetted cellulose sheets (wetted with an aqueous dispersion of highly dispersible PTFE) are saturated with a solution of caustic soda (or sodium hydroxide) and are allowed to steep for enough time for converting some of the cellulose into "soda cellulose" or the sodium salt of cellulose. During a process called "pressing," the soda cellulose is squeezed mechanically to remove excess caustic soda solution.

[0028] The soda cellulose is then mechanically shredded to increase the surface area and to make the cellulose more processible. This shredded cellulose is typically referred to as "white crumb." The white crumb undergoes "aging," whereby it is allowed to stand in contact with the oxygen of the ambient air, which allows the cellulose to partially oxidize (because of the high alkalinity of the white crumb) and become degraded to lower molecular weights. The degradation must be carefully controlled to produce chain lengths short enough to give manageable viscosities in the spinning solution, but still long enough to impart good physical properties to the solution spun fiber product.

[0029] After the above-described "aging" process, the white crumb is placed into a churn or some other mixing vessel and is treated with gaseous carbon disulfide (CS_2). In certain embodiments of the present invention, the incorporation of the aqueous dispersion

of the PTFE may occur just after the aging process, as the white crumb is being placed into the churn and being treated with CS_2. The soda cellulose reacts with the CS_2 to form xanthate ester groups. The CS_2 also reacts with the alkaline medium to form inorganic impurities, which give the cellulose mixture a characteristic yellow color. This material that results from the xanthation process is typically referred to as "yellow crumb."

[0030] Subsequently, the yellow crumb is dissolved in aqueous caustic solution. In certain embodiments of the present invention, the aqueous dispersion of highly dispersible PTFE may be added at this stage to the yellow crumb. The yellow crumb is not completely soluble at this stage. Specifically, because the cellulose xanthate solution (which is really a suspension) has such a high viscosity, it is typically termed the "viscose." The viscose is allowed to stand for a period of time to "ripen," and during ripening, two important processes occur: redistribution and loss of xanthate groups.

[0031] Next, the viscose is filtered to remove undissolved or undispersed materials that might disrupt the spinning process or cause defects in the rayon filament. In embodiments of the present invention where the highly dispersible PTFE is already incorporated into the viscose at this stage, this filtration step should be properly controlled so that the PTFE is not removed from the viscose. Additionally, bubbles of air entrapped in the viscose are removed prior to extrusion because they would cause voids or weak spots in the fine rayon filaments.

[0032] At this point, the fine filaments of PTFE-enhanced rayon are "solution spun." Specifically, the viscose is forced through a spinneret, a device resembling a shower head with many small holes. In certain embodiments of the present invention, the aqueous dispersion of highly dispersible PTFE is added to the viscose as the viscose is being

prepared to enter the spinneret. Each hole of the spinneret produces a fine filament of the viscose. As the viscose exits the spinneret, it comes in contact with a solution containing sulfuric acid, sodium sulfate, and usually Zn^{+2} ions. Several processes occur at this point, which cause the cellulose to be regenerated and to precipitate from the solution. Water diffuses out from the extruded viscose to increase the concentration in the filament beyond the limit of solubility. The xanthate groups form complexes with the Zn^{+2} ions, which draw the cellulose chains together. The acidic spin bath converts the xanthate functions into unstable xantheic acid groups, which spontaneously lose CS_2 and regenerate the free hydroxyls of cellulose. The result is the formation of PTFE-enhanced fine filaments of cellulose, or rayon.

[0033] After the PTFE-enhanced rayon filaments are formed, they are stretched while the cellulose chains remain relatively immobile. This causes the chains to stretch out and to orient along the axis of the fiber. As the chains become more parallel, interchain hydrogen bonds form, which gives the filaments the properties necessary for use as textile fibers. The PTFE-enhanced rayon fibers are then washed because freshly regenerated rayon contains many salts and other water-soluble impurities, which must be removed. The PTFE-enhanced rayon filaments are then cut if necessary. For example, if the PTFE-enhanced rayon is to be used as staple (*i.e.*, discreet lengths of fiber), the group of filaments, often termed "tow," is passed through a rotary cutter to provide a fiber, which can be processed in much the same way as a cotton fiber.

[0034] The PTFE-enhanced solution spun fibers produced by such a method may then be manufactured into a fabric or a textile, whereby the fabric or textile has the enhanced properties typically associated with the addition of PTFE to such articles. For example,

the fabric or textile exhibits a significantly decreased coefficient of friction, which may be useful in fabrics or textiles that are intended for apparel used in sports or recreational activities. Other properties of the solution spun fibers of the present invention include the exceptional wear resistance exhibited by the fibers.

[0035] Specifically, the solution spun fibers and/or the fabrics made from the fibers of the present invention may be wear tested to determine the wear resistance of the fibers. The wear testing may include Taber testing, Mace testing, and Pilling tests. Similarly, tests are performed to determine the tenacity of the fabric, the elongation of the fabric, and the draw. Generally, the full range of tests typically used to analyze the properties of a solution spun fiber may be employed to test the fibers of the present invention. Tests used in other industries and scientific test methods will also apply to the fibers and fabrics of the present invention.

[0036] The method and compositions of the present invention may be better understood through the working Examples detailed below. These Examples are intended to illustrate the invention and should not be construed as limiting the invention in any way.

EXAMPLES

Example 1: Production of PTFE-Enhanced Rayon Fiber:

[0037] In the present Example, PTFE-enhanced rayon fibers were made according to the method of the present invention and were compared to conventional rayon fibers. Specifically, three types of cellulose underwent initial testing to determine their usefulness in this Example: cosmetic cotton balls and two types of wooden cellulose. After both of the wooden cellulose pulp samples underwent steeping, these samples gave brown-colored matter because of the presence of lignin, hemicelluloses and other short-

chain carbohydrates. Conversely, initial testing of the cosmetic cotton balls resulted in a clean, orange-colored cellulose xanthate solution having the requisite high viscosity. Thus, only the cosmetic cotton ball raw material was used in further experiments.

[0038] The cosmetic cotton ball cellulose was mechanically shredded to increase the surface area and to make the cellulose easier to process. Then, the cellulose underwent steeping. Specifically, 1 gram of shredded cellulose ("Sample 1") was wet with 5 mL of water. Subsequently, 50 mL of a 15% sodium hydroxide solution was added under continuous stirring. The mixture was allowed to stand or steep for one hour. Visual observations of this mixture showed that the mixture looked like "white crumb" in a transparent liquid.

[0039] In a parallel experiment, 1 gram of the shredded cellulose ("Sample 2") was used, but instead of water, 5 mL of an aqueous dispersion of submicron PTFE powder was added to Sample 2. Specifically, the PTFE used in this Example is commercially available from Shamrock Technologies, Inc. under the trade name "NanoFLON W50C." As described for Sample 1 above, 50 mL of a 15% sodium hydroxide solution was then added to Sample 2 under continuous stirring, and the mixture was allowed to steep for one hour. Visual observations of the Sample 2 mixture showed that this mixture looked like "white crumb" in cloudy liquid.

[0040] Next, the soda cellulose of Sample 1 and the soda/PTFE cellulose of Sample 2 were each placed on a mesh filter and squeezed mechanically to remove any excess caustic soda solution. For Sample 1, the removed solution was slightly yellow and cloudy, whereas for Sample 2, the removed soda solution appeared more like a PTFE suspension.

[0041] Subsequently, 25 mL of CS_2 was added to each "white crumb" mixture under stirring, and each of Samples 1 and 2 were left under stirring for 3 hours. This process is generally known as xanthation. After 3 hours, the white crumb of Sample 1 changed in color to orange-yellow, and the matter was highly swollen in the transparent medium. For Sample 2, the CS_2 was white-yellow in color and not transparent. The excess of liquid was carefully removed from each of the samples, leaving behind what is typically termed as the "yellow crumb" for each of the two samples.

[0042] The next step that the samples underwent is typically termed dissolving. Specifically, 15 mL of a 15% NaOH solution was added to each yellow crumb gradually under stirring. After dissolving, Sample 1 resulted in a bright orange viscose solution, while Sample 2 resulted in a dim orange solution. The two viscoses produced by Samples 1 and 2 were allowed to stand for one hour to "ripen," and loose bubbles (that were introduced into each viscose by stirring) were removed through a degassing procedure.

[0043] Each viscose for Samples 1 and 2 was then ready to undergo a laboratory version of solution spinning. Specifically, a disposable syringe having a 1 mm diameter hole was used as a "spinneret" for spinning the rayon filaments. The viscose of Sample 1 was forced through the syringe into an acidic bath. Specifically the solution in this acidic bath contained 20% H_2SO_4 (sulfuric acid), 10% Na_2SO_4 (sodium sulfate), and 10% $ZnCl_2$ (zinc chloride). Subsequently, the viscose of Sample 2 was similarly forced through the syringe into an acidic bath.

[0044] Now that rayon filaments had been formed from each of Samples 1 and 2, the rayon filaments underwent "drawing." Specifically, the each of the rayon filaments was

stretched while the cellulose chains were still relatively mobile using two pairs of tweezers. Visual observations showed that the rayon filament formed from Sample 2 (the PTFE-enhanced rayon filament) stretched better than the filament made from Sample 1 that did not contain PTFE. Lastly, the freshly regenerated rayon filaments were washed with water several times.

[0045] Each of the rayon filaments was then analyzed using various laboratory techniques. First, the filaments underwent Fourier Transform Infrared Spectroscopy (FTIR) analysis, and the spectra showed that both were rayon fibers. For the rayon filament formed from Sample 2, its FTIR spectrum showed no distinctive PTFE peaks.

[0046] Next, each of the rayon filaments underwent differential scanning calorimetric (DSC) analysis. The DSC curves for the rayon filament formed from Sample 2 had a very small peak at 328.6°C at first heat and at 329.7°C at second heat. The values for ΔH were found to be 4.1 and 3.9 J/g, respectively. These values allowed the user to estimate the amount of PTFE in the rayon filaments of Sample 2 to be about 5% by weight.

[0047] Additionally, each of the rayon filaments underwent thermogravimetric (TGA) analysis, where the TGA curve for the rayon filament formed from Sample 2 showed the presence of PTFE to be 5.3%.

[redacted]

ABSTRACT OF THE DISCLOSURE

The present invention is directed to a method for making solution spun fibers having decreased coefficient of friction and other improved properties such as wear resistance and the like, when compared to conventional solution spun fibers. In the method of the present invention, polytetrafluoroethylene (PTFE) is incorporated into the fiber-forming substance during the solution spinning process before passing through the spinneret. PTFE that is useful in the present invention includes PTFE powder that is dispersible to low micron or submicron particle size and aqueous or organic dispersions of such highly dispersible PTFE powder. The present invention is also directed to fabrics, textiles, and other articles of manufacture formed from the PTFE-enhanced solution spun fibers of the present invention.

[redacted]